

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION FOR UNITED STATES LETTERS PATENT

INVENTION:

SUSPENSIONS OF PARTICLES IN NON-AQUEOUS SOLVENTS

INVENTOR:


WILLIAM FRANKLIN HARRIS, JR.

CERTIFICATE OF EXPRESS MAIL

I hereby certify that foregoing documents are being deposited with the United States Postal Service as Express Mail, postage prepaid, in an envelope addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231.

Express Mail No. EL 728799960 US

Date 13 July 2001


Autumn L. Walker

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of application number 09/771,226, which was filed January 26, 2001.

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to stable concentrated non-aqueous suspensions of particles with excellent storage stability. More specifically, the particle suspensions are characterized by having a medium of low molecular weight polyalkylene glycol and a stabilizer of a hydrogenated castor wax.

2. Description of the Related Art

Heretofore, in preparing aqueous dilutions of particles, it has been necessary to utilize solid ingredients that are mixed with water or other aqueous fluid at the job site. A number of disadvantages are inherent in such mixing procedures, particularly when large volumes of solutions are prepared. For example, special mixing equipment for mixing the dry additives with water is required and problems such as chemical dusting, uneven mixing, and extended preparation and mixing time are involved. In addition, the mixing and physical handling of large quantities of dry chemicals require a great deal of manpower, and, where continuous mixing is required, the accurate and efficient handling of chemicals is extremely difficult.

In addition, when particles are used, they are typically added to water to make a dilute solution, resulting in the generation of fugitive dust. This dust has a number of potential detrimental effects. Workers preparing the solution can inhale the dust, and some of the particles that can be suspended in a non-toxic solvent produce dust when handled in a powdered form.

This may produce a respiratory allergenic response, irritation, or other toxic effect when inhaled.

Dust can also drift to areas where it is not intended.

In the agricultural industry, many handling problems may arise when one is forced to prepare aqueous end-use formulations and/or slurries from solids, especially active solids, e.g. wettable bioactive powders. Farmers preparing tank mixes of herbicides, insecticides and/or other bioactives from solids for applications to crops and soil are exposed to certain safety hazards and inconveniences due to the generation of noxious dusts which may be irritable to the skin and hazardous to breathe.

Additionally, finely ground powders, even so-called powders, of many water-soluble bioactives do not disperse well when prepared as tank mixes. They have poor spontaneity or "bloom" and have low suspendability. They have poor re-dispersibility and are incompatible with other bioactives as compared to liquid bioactive concentrates. Thus, final formulators, such as farmers, when preparing diluted aqueous active compositions find that the handling and application of solids materials, such as fertilizers, are much easier if the material can be supplied in a fluid rather than solid form. Economics then dictate that the active material be supplied in a highly concentrated fluid to the final formulator.

Saturation solubility in water of many water-soluble active constituents, such as potassium chloride, is too low to make it economical for supply to the end-user simply in the form of a solution. Alternatively, highly concentrated suspensions of water-soluble compounds, both in water and in organic liquids, have very poor storage, freeze/thaw, and heat/cool stability. As a result of the spontaneous crystal dissolution-recrystallization process, there occurs a progressive increase in the size of the particulate active material. This increase in particle size results in settling, bleed and changes in visco-elastic properties and thus severely limits

concentrate loading levels.

To avoid lump or dust formation and its associated problems, the particles can be added to the aqueous systems as liquid slurries or suspensions. There currently exists a number of methods for accomplishing this, and the compositions prepared thereby. These methods often employ use oil carriers (e.g., mineral, isoparaffin or diesel) to suspend and deliver the particles to the aqueous systems. In applications where the materials may be used in off-shore oil well treatment fluids which may be eventually discharged into the environment, recent regulations by the Environmental Protection Agency limit the amount of oil or grease that can be used in offshore oilfield applications for well treatment fluids. The National Pollutant Discharge Elimination System (NPDES) General Permit issued on April 19, 1999 (Federal Register Vol. 64 No. 74) limits the oil and grease to a daily maximum concentration of 42 mg/l and a monthly average of 29 mg/l when the suspension is diluted to the intended use level with fresh or salt water. Unlike the liquid suspensions that contain diesel fuel or other hydrocarbon solvents the suspensions of the present invention contain minimal detectable oil or grease when diluted to the concentration appropriate for well treatment.

U.S. Patent No. 5,091,448 discloses a suspending medium for a water-soluble polymer, while U.S. Patent No. 5,631,313 discloses a suspending medium for particles. These two patents utilize isoparaffin oils as the solvent for the suspensions where a styrene/isoprene copolymer is used as the suspension agent. Upon dilution to the intended use concentration in fresh or salt water for a well treatment fluid, the dilution contains a much higher concentration of oil and grease than is permitted by the above regulations. Furthermore the styrene/isoprene copolymer that is used to stabilize the suspension is insoluble in water miscible solvents such as the polyalkylene glycols of the present invention.

U.S. Patent No. 5,925,182 discloses a stable liquid suspension composition including a liquid carrier, a solid fatty acid or a salt thereof, and a solid particulate wherein the liquid carrier is selected from the group consisting of oils, olefins, terpenes, glycols, esters, ethers, alcohols, and combinations of any two or more thereof and the liquid carrier, solid fatty acid or salt thereof, and solid particulate are each present in the composition in a stabilizing amount sufficient to produce a stable liquid suspension. Also disclosed is a stable liquid composition including a liquid carrier, an oil soluble polymer, and a solid particulate wherein the liquid carrier is selected from the group consisting of olefins, terpenes, esters, and combinations of any two or more thereof and the liquid carrier, oil-soluble polymer, and solid particulate are each present in the composition in a stabilizing amount sufficient to effect the formation of a stable liquid suspension. Although this patent includes some solvents that may be environmentally friendly and some that contribute to oil and grease as measured by the EPA method, the suspension agent is based on a fatty acid or salt thereof, or an oil soluble polymer, either of which will be measured as oil and grease.

In addition to the oil carrier fluid, many hydrocarbon solvent based slurries usually contain clay or clay like particulates that act to viscosify and stabilize the non-aqueous suspension. The clay component itself is also often times an undesirable component. This is particularly true in oil and gas field applications where incorporation of the clay into the slurries, which is necessary to keep the particles in suspension, impairs the permeability of the oil or gas bearing strata. This is the very same problem caused by the formation of lumps that the oil suspension or slurry is supposed to eliminate.

Many aqueous suspensions include a variety of inorganic and organic particles that use water as the continuous phase for preparing the liquid solution or suspension. While the use of

water is certainly environmentally acceptable and reduces the dusting properties of many solid particles, its use is counterproductive with many solids. Among these are solids that may be reactive with water. Also, particles may be wholly or partially soluble in water and this solubility may limit the maximum concentration of the dispersed phase that can be incorporated into an aqueous suspension. The use of a non-solvent for suspending certain solids results in a controlled release of the solids because the particles must first dissolve into water before they become functional.

U.S. Patent No. 4,673,526 discloses an anhydrous skin cleansing composition containing an oil phase, an emulsifying agent, and particulate water soluble polymeric abrasive particles. This compound contains an oily phase, at least one emulsifying agent, and at least one abrasive substance. The compound is presented in anhydrous form and the abrasive substance in suspension in the oily phase is highly hydrosoluble with an average particle size between 50 and 1000 microns. This compound allows the deep cleansing of the skin through exfoliant action.

U.S. Patent No. 5,985,252 discloses a suspension antiperspirant composition for topical application to the human skin including from 10 to 26% by weight of the composition of a solid particulate antiperspirant active suspended in a cosmetic base. The antiperspirant active includes a blend of an antiperspirant active with relatively small particles with a volume average particle size in the range of from 0.5 to 8 micrometers and an antiperspirant active with relatively large particles having a volume average particle size in the range of larger than 12 to smaller than 50 micrometers with the weight ratio of the antiperspirant active having smaller particles to the antiperspirant active having larger particles in the composition is in the region of 5:1 to 1:5 by weight.

U.S. Patent No. 5,863,647 discloses a monodisperse glycol suspension having excellent dispersion stability at a pH within a wide range. The suspension includes a monodisperse suspension in a glycol of spherical fine particles of an amorphous inorganic oxide having an average particle diameter of 0.15 to 5 micrometers and a relative particle size standard deviation of 1.0 to 1.5 and containing glycol bonded to its surface in amounts of 0.003 to 5 millimoles glycol, per gram of fine particles. This monodisperse suspension is useful as a raw material for the production of a polyester film having improved slipperiness.

Despite the above teachings, there still exists a need for liquid suspensions for water-soluble polymers that are environmentally friendly; suitable for use in personal care products, such as cosmetics and shampoos and the like; can be manufactured using ingredients suitable for use in indirect contact with food or as a pesticide adjuvant; are extremely stable over long periods of time and are operative over a wide temperature range; and are comprised of materials that are commercially available or easy to manufacture.

11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60
61
62
63
64
65
66
67
68
69
70
71
72
73
74
75
76
77
78
79
80
81
82
83
84
85
86
87
88
89
90
91
92
93
94
95
96
97
98
99
100
101
102
103
104
105
106
107
108
109
110
111
112
113
114
115
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
131
132
133
134
135
136
137
138
139
140
141
142
143
144
145
146
147
148
149
150
151
152
153
154
155
156
157
158
159
160
161
162
163
164
165
166
167
168
169
170
171
172
173
174
175
176
177
178
179
180
181
182
183
184
185
186
187
188
189
190
191
192
193
194
195
196
197
198
199
200
201
202
203
204
205
206
207
208
209
210
211
212
213
214
215
216
217
218
219
220
221
222
223
224
225
226
227
228
229
230
231
232
233
234
235
236
237
238
239
240
241
242
243
244
245
246
247
248
249
250
251
252
253
254
255
256
257
258
259
260
261
262
263
264
265
266
267
268
269
270
271
272
273
274
275
276
277
278
279
280
281
282
283
284
285
286
287
288
289
290
291
292
293
294
295
296
297
298
299
300
301
302
303
304
305
306
307
308
309
310
311
312
313
314
315
316
317
318
319
320
321
322
323
324
325
326
327
328
329
330
331
332
333
334
335
336
337
338
339
340
341
342
343
344
345
346
347
348
349
350
351
352
353
354
355
356
357
358
359
360
361
362
363
364
365
366
367
368
369
370
371
372
373
374
375
376
377
378
379
380
381
382
383
384
385
386
387
388
389
390
391
392
393
394
395
396
397
398
399
400
401
402
403
404
405
406
407
408
409
410
411
412
413
414
415
416
417
418
419
420
421
422
423
424
425
426
427
428
429
430
431
432
433
434
435
436
437
438
439
440
441
442
443
444
445
446
447
448
449
450
451
452
453
454
455
456
457
458
459
460
461
462
463
464
465
466
467
468
469
470
471
472
473
474
475
476
477
478
479
480
481
482
483
484
485
486
487
488
489
490
491
492
493
494
495
496
497
498
499
500
501
502
503
504
505
506
507
508
509
510
511
512
513
514
515
516
517
518
519
520
521
522
523
524
525
526
527
528
529
530
531
532
533
534
535
536
537
538
539
540
541
542
543
544
545
546
547
548
549
550
551
552
553
554
555
556
557
558
559
560
561
562
563
564
565
566
567
568
569
570
571
572
573
574
575
576
577
578
579
580
581
582
583
584
585
586
587
588
589
590
591
592
593
594
595
596
597
598
599
600
601
602
603
604
605
606
607
608
609
610
611
612
613
614
615
616
617
618
619
620
621
622
623
624
625
626
627
628
629
630
631
632
633
634
635
636
637
638
639
640
641
642
643
644
645
646
647
648
649
650
651
652
653
654
655
656
657
658
659
660
661
662
663
664
665
666
667
668
669
670
671
672
673
674
675
676
677
678
679
680
681
682
683
684
685
686
687
688
689
690
691
692
693
694
695
696
697
698
699
700
701
702
703
704
705
706
707
708
709
710
711
712
713
714
715
716
717
718
719
720
721
722
723
724
725
726
727
728
729
730
731
732
733
734
735
736
737
738
739
740
741
742
743
744
745
746
747
748
749
750
751
752
753
754
755
756
757
758
759
760
761
762
763
764
765
766
767
768
769
770
771
772
773
774
775
776
777
778
779
780
781
782
783
784
785
786
787
788
789
790
791
792
793
794
795
796
797
798
799
800
801
802
803
804
805
806
807
808
809
810
811
812
813
814
815
816
817
818
819
820
821
822
823
824
825
826
827
828
829
830
831
832
833
834
835
836
837
838
839
840
841
842
843
844
845
846
847
848
849
850
851
852
853
854
855
856
857
858
859
860
861
862
863
864
865
866
867
868
869
870
871
872
873
874
875
876
877
878
879
880
881
882
883
884
885
886
887
888
889
890
891
892
893
894
895
896
897
898
899
900
901
902
903
904
905
906
907
908
909
910
911
912
913
914
915
916
917
918
919
920
921
922
923
924
925
926
927
928
929
930
931
932
933
934
935
936
937
938
939
940
941
942
943
944
945
946
947
948
949
950
951
952
953
954
955
956
957
958
959
960
961
962
963
964
965
966
967
968
969
970
971
972
973
974
975
976
977
978
979
980
981
982
983
984
985
986
987
988
989
990
991
992
993
994
995
996
997
998
999
1000

SUMMARY OF THE INVENTION

5013
21

In accordance with the present invention, a non-aqueous suspension includes solid particles, polyalkylene glycol, and a suspension stabilizer of a hydrogenated castor oil or wax. The non-aqueous suspension includes the solid particles in an amount between about 0.1 and about 75 percent by weight of the suspension, the polyalkylene glycol in an amount between about 24 and about 99.8 percent by weight of the suspension, and the suspension stabilizer in an amount between about 0.1 and about 5.0 percent by weight of the suspension. The non-aqueous suspension may further include one or more of the following additive materials: proppants, antifoaming agents, surfactants, corrosion inhibitors, pH buffers, and preservatives.

The polyalkylene glycol includes polyethylene glycol, polypropylene glycol, ethylene oxide propylene oxide block copolymers, and mixtures thereof. The polyalkylene glycol may include between about 0.1 and 4% by weight of the polyalkylene glycol of a thickener including partially neutralized polyacrylic acid, hydroxypropyl cellulose, highly substituted hydroxypropyl guar, fumed silica, hydrophobic silica, and mixtures thereof.

The solid particles include non-polymeric particles that are either inorganic particles or organic particles. The inorganic particles include boron compounds; alkaline earth peroxides; magnesium peroxide or calcium peroxide; iron oxide; calcium aluminate, calcium carbonate, magnesium carbonate, calcium oxide, magnesium oxide, calcium hydroxide and magnesium hydroxide and mixtures thereof; and siliceous or ceramic particles. The organic particles include gilsonite; lignosulfonates and the sodium, potassium, ammonium, calcium and magnesium salts thereof; and ethylenediaminetetraacetic acid and the salts thereof. The particles further include fertilizers selected from the group consisting of potassium nitrate, ammonium dihydrogenphosphate, ammonium nitrate, sodium nitrate ammonium phosphate, ammonium

polyphosphate, potassium hydrogen phosphate, disodium hydrogen phosphate, urea, and mixtures thereof. The particles still further include pesticides selected from the group consisting of boric acid, butocarboxime, acephate, dimethoate, dimehypo, vamidothion, methomyl and mixtures thereof. The particles even further include herbicides selected from the group consisting of dalapon (2,2 dichloropropirionic acid, sodium salt) ammonium sulfamate, dicamba, cacodylic acid, fomesafen; glyphosate and mixtures thereof. The particles yet further include fungicides selected from the group consisting of copper sulfate, fosetyl-Al aluminum tris (O-ethyl phosphonate), benalaxyl, guazatine, kasugamycin and mixtures thereof.

In a method of formulating a non-aqueous suspension, solid particles from about 0.1 to about 75% suspension weight and a hydrogenated castor wax or oil from about 0.1 to about 5.0% suspension weight are dispersed into from about 24 to about 99.8% suspension weight of polyalkylene glycol. The solid particles, hydrogenated castor wax, and polyalkylene glycol are mixed until the solid particles are uniformly dispersed in the polyalkylene glycol and the hydrogenated castor wax has developed desired suspension properties.

It is therefore an object of the present invention to provide non-aqueous suspensions that are environmentally friendly.

It is another object of the present invention to provide non-aqueous suspensions suitable for use in personal care products, such as cosmetics, shampoos, and the like.

It is still another object of the present invention to provide non-aqueous suspensions that can be manufactured using ingredients suitable for use in indirect contact with food or as a pesticide adjuvant.

It is a further object of the present invention to provide non-aqueous suspensions that are extremely stable over long periods of time and that are operative over a wide temperature range.

It is still a further object of the present invention to provide non-aqueous suspensions that are comprised of materials that are commercially available or easy to manufacture.

Still other objects, features, and advantages of the present invention will become evident to those of ordinary skill in the art in light of the following.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

A first element of a liquid particle suspension includes solid particles in an amount from 0.1 to 75% of the weight of the suspension. The particles are typically non-polymeric and may be inorganic or organic. Typically the particles of interest will have one or more of the following characteristics that favor use in a concentrated non-aqueous dispersion: 1) low water solubility or materials with high water solubility that have a tendency to have very poor storage, freeze/thaw, and heat/cool stability; 2) the need for a controlled dissolution rate; 3) materials that are reactive with water or a water hydratable agent; 4) materials that cause nuisance dusts; and 5) materials that are easier to meter in liquid form rather than by weighing and pre-dissolving in water when needed at a job site. Typically the particles will have an average particle size between 0.1 micron and 500 microns. More preferably, the particles will have an average particle size between 1 micron and 300 microns. Most preferably the particles will have an average particle size between 5 microns and 200 microns.

Solid particles include a wide variety of materials including inorganic as well as organic solid particles. Illustrative examples of specific solid particles include but are not limited to: peroxides such as magnesium peroxide and calcium peroxide, magnesium oxide, calcium oxide, herbicides including butocarboxime, acephate, dimethoate, dimehypo, vamidothion, methomyl, dalapon (2,2 dichloropropionic acid, sodium salt) ammonium sulfamate, dicamba, cacodylic acid, fomesafen; glyphosate, copper sulfate, fosetyl-Al aluminum tris (O-ethyl phosphonate),

benalaxyl, guazatine, kasugamycin; insecticides, sulfonated asphalt, salts of sulfonated asphalt, lime, sodium bicarbonate, sodium carbonate, sodium borate, boric acid, potassium nitrate, ammonium dihydrophosphate, ammonium nitrate, sodium nitrate ammonium phosphate, ammonium polyphosphate, potassium hydrogen phosphate, disodium hydrogen phosphate, urea, molybdenum disulfide, pigments, activated carbon, carbon black, unintahite (gilsonite), graphite, iron, iron oxide, zinc, tin, quebracho, lignin, lignite, causticized lignite, lignosulfonate, chrome lignosulfonate, naphthalenesulfonate; and, combinations of two or more thereof.

A second element of the suspension includes polyalkylene glycol or thickened polyalkylene glycol. The amount of this ingredient varies between about 24 to 99.8% of the weight of the suspension. Particularly preferred are polyethylene glycol, polypropylene glycol or ethylene oxide propylene oxide block copolymers. Most preferred is low molecular weight glycols having a molecular weight of less than 1000, more preferably having a molecular weight between 100 and 600 and most preferably between 200 and 500. Polyethylene glycol having a molecular weight of 200 can also be used, for example. Polyethylene or polypropylene glycol having a molecular weight of 300 or higher and manufactured in accordance with the specifications of the National Formulary can be used in cosmetic grade applications. A technical grade of polyethylene or polypropylene glycol having a molecular weight of 300 or higher as indirect additives for food contact materials and the like may also be used. Technical grades of polyethylene glycol with a molecular weight of 300 or higher are exempt from residue tolerance when used as inert ingredients in pesticide formulations employed in growing crops.

The term "thickened polyalkylene glycol" refers to polyalkylene glycols having a thickener preferably between 0.1 and 4% by weight of the polyalkylene glycol selected from the group consisting of partially neutralized polyacrylic acid, hydroxypropyl cellulose, highly

substituted hydroxypropyl guar, hydrated thickening silica including fumed silica and hydrophobic fumed silica, or their functional equivalents or mixtures thereof. The preferred hydrated thickening silicas, also known as thickening silicas, are colloidal gel silicas or hydrophobic derivatives thereof. More preferred ones are Aerosil[®]200 silica, available from Degussa Corporation, Ridgefield Park NJ, and CAB-O-SIL[®]M-5 and TS-530 available from Cabot Corporation, Tuscola, IL. The most preferred is CAB-O-SIL[®] TS-530.

A third element of the suspension includes a finely divided hydrogenated oil or wax. Most preferably this hydrogenated oil or wax is hydrogenated castor wax. This material is present in the amount from 0.1 to 5% of the weight of the suspension. More preferably in the amount of 0.3 to 3% of the weight of the suspension. Most preferably in the amount of 0.5 to 2% of the weight of the suspension. The preferred hydrogenated castor wax is sold by Süd Chemie of Louisville, Kentucky under the name of Rheocin[®]. Rheocin[®] is acceptable for use as an indirect food additive in Title 21 of the Code of Federal Regulations.

In addition to the foregoing three elements, the suspension may also contain optional ingredients such as: antifoaming agents, corrosion inhibitors, preservatives, surfactants, water miscible co-solvents, and other materials that aid in the performance of the solid particles in their intended applications.

The suspensions may be used in any number of commercial applications where dry solid particles have previously been used, as well as in applications where solid particles have not been well suited due to their undesirable physical properties, such as low water solubility, stability in concentrated aqueous solutions, the need for a controlled dissolution rate, materials that are reactive with water or a water hydratable suspension agent, materials that cause nuisance

dusts, and materials that are easier to meter in liquid form rather than by weighing and pre-dissolving in water when needed at a job site.

The suspensions are particularly useful for applications involving dispersing solid particles in aqueous solutions. Included among such applications are the following: environmental applications (e.g., remediation projects), agricultural applications metal working fluids, paper applications, textile applications, cosmetic or personal care applications, cleaners, detergents, application of pesticides, aerial firefighting applications, construction products (e.g. paint, joint cements, texture finishing compounds and the like), emulsion stabilizers, adhesives, inks, and oil field applications.

EXAMPLES

Example 1 - Inventive Suspension Media

A method for making the suspensions of solid particles includes dispersing from 0.1 to 75% suspension weight of a particle and from 0.1 to 5.0% suspension weight of a hydrogenated castor wax or oil into from 24 to 99% suspension weight of polyalkylene glycol. The solid particles, hydrogenated castor wax, and polyalkylene glycol are mixed using conventional agitation, such as an overhead mixer, until the solid particles are uniformly dispersed in the polyalkylene glycol and the hydrogenated castor wax has developed the desired suspension properties. Desirable properties of the suspension include but are not limited to the following:

1. The solvent and suspension stabilizer are environmentally friendly and non-toxic;
2. The particle suspension does not create dust upon addition to water;
3. The suspension remains stable for extremely long periods of time exhibiting minimum separation of solvent and particulate and no packing of the solid particles;
4. The suspension is easily pourable or pumpable;

5. The suspended particles disperse in water better than if the solid is added to water;

Example 2 – Sodium Tetraborate Inventive Suspension

810 grams of a powdered anhydrous sodium tetraborate and 20 grams of Rheocin[®] are dispersed into 1160 grams of pre-thickened polyethylene glycol (200MW) containing 10 grams thickening silica, specifically CAB-O-SIL[®] TS-530, in a 2000 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition (% by weight)	
Sodium tetraborate	40.50%
Rheocin [®]	1.0%
Pre-thickened Polyethylene glycol 200MW	58.4%
Initial viscosity	4610 cP
Density	1.365 g/ml
Pounds of sodium tetraborate per U.S. Gallon	4.6

Properties upon aging

	Separation	Packing
24 hours	0%	None
3 days	0%	None
1 month	1%	None
3 months	4%	None

The above composition is easily pourable or pumpable.

Example 3 - Sodium Tetraborate Control Suspension

This example compares the suspension properties of anhydrous sodium tetraborate to Example 2 without the use of the hydrogenated castor wax. 810 grams of sodium tetraborate is dispersed into 1170 grams of polyethylene glycol (200MW) in a 2000 ml beaker. The mixture is

agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition (% by weight)	
Sodium tetraborate	40.50%
Polyethylene glycol 200MW	59.50%
Initial viscosity	1250 cP
Density	1.37 g/ml
Pounds per Gallon of Borate Compound	4.6

Properties on aging	<u>Supernatant separation</u>	<u>Particle packing</u>
24 hours	12% by volume	Medium packed Difficult to remix with stirring rod
3 days	22% by volume	Hard packed can not remix with stirring rod
1 week	30% by volume	Hard packed can not remix with stirring rod

Example 4 – Magnesium Peroxide Inventive Suspension

250 grams of a powdered Magnesium Peroxide and 5.0 grams of Rheocin[®] are dispersed into 240 grams pre-thickened polyethylene glycol (200MW) containing 5 grams thickening silica, specifically CAB-O-SIL[®] TS-530a making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement

of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition	
Magnesium Peroxide	50%
Rheocin®	1%
Pre-thickened Polyethylene glycol 200MW	49%
Initial Viscosity	
	3200 cP
Density	1.515 g/ml
Pounds of magnesium peroxide per U. S. Gallon	6.3

Properties on aging		
	Separation	Packing
24 hours	0%	None
3 days	0%	None
1 month	1%	None
3 months	2%	None

The above compositions are easily pourable or pumpable

Example 5 Inventive Colemanite Suspension

Turkish Colemanite is an ore rich in calcium borate supplied by American Borate. 240 grams of a powdered Colemanite and 4.5 grams of Rheocin® are dispersed into 207.5 grams pre-thickened polyethylene glycol (200MW) containing 4.5 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Colemanite	53.1%
Rheocin®	1%

Pre-thickened Polyethylene glycol 200MW	45.9%
Initial Viscosity	2700 cP
Initial density	1.6 g/ml
Pounds of Colemanite per U. S. gallon	7.1

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	1%	None
3 months	2%	None

The above compositions are easily pourable or pumpable

Example 6 – Colemanite Control Suspension

This example compares the suspension properties of Colemanite to example 5 without the use of the hydrogenated castor wax suspension agent. 240 grams of a powdered Colemanite and are dispersed into 212 grams pre-thickened polyethylene glycol (200MW) containing 4.5 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Colemanite	53.1%
Pre-thickened Polyethylene glycol 200MW	46.9%
Initial Viscosity	1100 cP
Initial density	1.6 g/ml
Pounds of Colmanite per U. S. gallon	7.1

Properties on aging

Separation	Packing
------------	---------

24 hours	2%	None
7 days	22%	Significant settling; difficult to remix
1 month	27%	Hard packed sediment; very difficult to remix
3 months	29%	Hard packed sediment; very difficult to remix

Example 7 – Inventive Gilsonite Suspension

Gilsonite is an asphaltic material or solidified hydrocarbon used in a variety of applications in explosives, oil field and other industrial applications. Powdered gilsonite is available from American Gilsonite and others. 100 grams of a powdered gilsonite and 6 grams of Rheocin[®] are dispersed into 194 grams polyethylene glycol (200MW) making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Powdered gilsonite	33.3%
Rheocin [®]	2%
Polyethylene glycol 200MW	64.7%
Initial Viscosity	3300 cP
Initial density	1.1 g/ml
Pounds of gilsonite per U. S. gallon	3

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	0%	None
3 months	1%	None

The above compositions are easily pourable or pumpable

Example 8 – Control Gilsonite Suspension

This example compares the suspension properties of gilsonite to example 7 without the use of the hydrogenated castor wax suspension agent. 100 grams of a powdered gilsonite and are dispersed into 200 grams polyethylene glycol (200MW) making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:		% by weight
Powdered Gilsonite		33.3%
Polyethylene glycol 200MW		66.7%
Initial Viscosity		360 cP
Initial density		1.1 g/ml
Pounds of Gilsonite per U. S. gallon		7.1
Properties on aging		
	Separation	Packing (Floating)
24 hours	19%	Note: Due to the light density of the suspended phase (gilsonite) separation rose to the surface in this case (floated). At 24 hrs it was able to be remixed with some difficulty.
7 days	22%	Significant floating; difficult to remix
1 month	33%	The individual particles of gilsonite have coalesced into a continuous phase. This phase solid and can not be remixed without extreme measures.
3 months	35%	The overall appearance is unchanged from 1 month. The coalesced upper phase has formed a tar like continuous phase which can not be remixed without extreme measures.

Example 9 – Inventive Calcium Aluminate Suspension

Calcium aluminate is an alkaline pH buffer supplied by Sintertec Division of BPI, Inc. It has a low solubility in water, which makes it useful as a controlled release buffer that supplies alkalinity as acid enters a system. The low water solubility of calcium aluminate makes it difficult to supply as an aqueous dispersion of solution. 275 grams of a powdered calcium aluminate and 8 grams of Rheocin[®] are dispersed into 196 grams pre-thickened polyethylene glycol (200MW) containing 4 grams thickening silica, specifically CAB-O-SIL[®] TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Calcium aluminate	57.4%
Rheocin [®]	1.7%
Pre-thickened Polyethylene glycol 200MW	40.1%
Initial Viscosity	4950 cP
Initial density	1.9 g/ml
Pounds of calcium aluminate per U. S. gallon	9.1

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	1.5%	None
3 months	3%	None

The above compositions are easily pourable or pumpable

Example 10 – Control Calcium Aluminate Suspension

This example compares the suspension properties of calcium aluminate to example 9 without the use of the hydrogenated castor wax suspension agent. 275 grams of a powdered calcium aluminate is dispersed into 196 grams pre-thickened polyethylene glycol (200MW) containing 4 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight	
Calcium aluminate	57.4%	
Pre-thickened Polyethylene glycol 200MW	42.6%	
Initial Viscosity	700 cP	
Initial density	1.85 g/ml	
Pounds of Calcium Aluminate per U. S. gallon	8.9	
Properties on aging	Separation	Packing
24 hours	2%	None
7 days	17%	Significant settling; difficult to remix
1 month	23%	Hard packed sediment; very difficult to remix

Example 11 – Inventive Calcium Carbonate Suspension

Calcium carbonate is widely used in a number of industries as an extender pigment. It is also used in agricultural applications as a pH buffer to adjust the pH of acidic soils. It has a low solubility in water. The low water solubility of calcium carbonate makes it difficult to supply as an aqueous solution. 200 grams of a powdered calcium carbonate and 8 grams of Rheocin® are

dispersed into 192 grams pre-thickened polyethylene glycol (200MW) containing 4 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Calcium carbonate	50%
Rheocin®	2%
Pre-thickened Polyethylene glycol 200MW	48%
Initial Viscosity	2700 cP
Initial density	1.57 g/ml
Pounds of calcium carbonate per U. S. gallon	6.5

Properties on aging	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	2%	None
3 months	3%	None

The above compositions are easily pourable or pumpable

Example 12 – Control Calcium Carbonate Suspension

This example compares the suspension properties of calcium carbonate to example 11 without the use of the hydrogenated castor wax suspension agent. 200 grams of a powdered calcium carbonate is dispersed into 200 grams pre-thickened polyethylene glycol (200MW) containing 4 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20

rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition: % by weight

Calcium carbonate	50%
Pre-thickened Polyethylene glycol 200MW	50%

Initial Viscosity	280 cP
Initial density	1.58 g/ml
Pounds of calcium carbonate per U. S. gallon	6.5

Properties on aging

	Separation	Packing
24 hours	12%	None
7 days	43%	Hard packed sediment; very difficult to remix
1 month	45%	Hard packed sediment; very difficult to remix
3 months	45%	Hard packed sediment; very difficult to remix

Example 13 – Inventive Iron Oxide Suspension

Iron (III) oxide (Fe_2O_3) is used as a pigment, as a mordant, as a catalyst and on magnetic recording tapes. It is also used as a weighting agent in oil field applications. The low water solubility of Fe_2O_3 makes it difficult to supply as an aqueous solution. 390 grams of a Iron (III) oxide which has an average particle size of 5 micrometers and 6 grams of Rheocin[®] are dispersed into 350 grams pre-thickened polyethylene glycol (200MW) containing 6 grams thickening silica, specifically CAB-O-SIL[®] TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement

of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition: % by weight

Fe ₂ O ₃	51.9%
Rheocin®	0.8%
Pre-thickened Polyethylene glycol 200MW	47.3%

Initial Viscosity	6100 cP
Initial density	1.8 g/ml
Pounds of Fe ₂ O ₃ per U. S. gallon	7.8

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	0%	None
3 months	2%	None

The above compositions are easily pourable or pumpable

Example 14 – Control Iron Oxide Suspension

This example compares the suspension properties of Fe₂O₃ to example 13 without the use of the hydrogenated castor wax suspension agent. 390 grams of a Fe₂O₃ having an average particle size of 5 micrometers is dispersed into 356 grams pre-thickened polyethylene glycol (200MW) containing 6 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition: % by weight

Fe ₂ O ₃	51.9%
Pre-thickened Polyethylene glycol 200MW	48.1%

Initial Viscosity	3000 cP
Initial density	1.8 g/ml
Pounds of Fe ₂ O ₃ per U. S. gallon	7.8

Properties on aging

	Separation	Packing
24 hours	<1%	None
7 days	<1%	None
1 month	5%	Slight packing, easy to remix
3 months	15%	Moderate packing, fairly easy to remix

Example 15 – Inventive Titanium Dioxide Suspension

Titanium dioxide (TiO₂) is used as a primary pigment in a variety of coating applications. The low water solubility of TiO₂ makes it difficult to supply as an aqueous solution. 250 grams of a TiO₂ which has been sifted through a 325 mesh screen and 5 grams of Rheocin® are dispersed into 370 grams pre-thickened polyethylene glycol (200MW) containing 5 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
TiO ₂	40%
Rheocin®	0.8%
Pre-thickened Polyethylene glycol 200MW	59.2%
Initial Viscosity	4200 cP
Initial density	1.57 g/ml
Pounds of TiO ₂ per U. S. gallon	5.2

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	0%	None
3 months	<1%	None

The above compositions are easily pourable or pumpable

Example 16 – Control Titanium Dioxide Suspension

This example compares the suspension properties of TiO₂ to example 15 without the use of the hydrogenated castor wax suspension agent. 250 grams of a TiO₂ which had been sifted through a 325 mesh screen is dispersed into 370 grams pre-thickened polyethylene glycol (200MW) containing 5 grams thickening silica, specifically CAB-O-SIL[®] TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition: % by weight

TiO ₂	40%
Pre-thickened Polyethylene glycol 200MW	60%
Initial Viscosity	2100 cP
Initial density	1.57 g/ml
Pounds of TiO ₂ per U. S. gallon	5.2

Properties on aging

	Separation	Packing
24 hours	<1%	None
7 days	<1%	None
1 month	7%	Slight packing, fairly easy to remix

3 months 13% Moderate packing; fairly easy to remix

Example 17 – Inventive Calcium Lignosulfonate Suspension

Calcium lignosulfonate is widely used in a number of industries, including oil well cements, as a dispersant. It has a low solubility in water. The low water solubility of calcium lignosulfonate makes it difficult to supply as an aqueous solution. 105 grams of a powdered calcium lignosulfonate and 3 grams of Rheocin[®] are dispersed into 192 grams pre-thickened polyethylene glycol (200MW) containing 3 grams thickening silica, specifically CAB-O-SIL[®] TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
Calcium lignosulfonate	35%
Rheocin [®]	1%
Pre-thickened Polyethylene glycol 200MW	64%
Initial Viscosity	1800 cP
Initial density	1.13 g/ml
Pounds of calcium lignosulfonate per U. S. gallon	3.3

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	0%	None
3 months	0%	None

The above compositions are easily pourable or pumpable

Example 18 – Control Calcium Lignosulfonate Suspension

This example compares the suspension properties of calcium lignosulfonate to example 17 without the use of the hydrogenated castor wax suspension agent. 105 grams of a powdered calcium lignosulfonate is dispersed into 195 grams polyethylene glycol (200MW) making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
--------------	-------------

Calcium lignosulfonate	35%
Polyethylene glycol 200MW	65%

Initial Viscosity	860 cP
Initial density	1.13 g/ml
Pounds of calcium lignosulfonate per U. S. gallon	3.3

Properties on aging

	Separation	Packing
24 hours	5%	Separation in the middle of the column easy to remix
7 days	24%	Triphase separation. Upper phase crusty and sticky, middle phase is clear liquid, bottom phase is hard packed; difficult to remix
1 month	24%	Triphase separation. Upper phase crusty and sticky, middle phase is clear liquid, bottom phase is hard packed; difficult to remix
3 months	26%	Triphase separation. Upper phase crusty and sticky, middle phase is clear liquid, bottom phase is hard packed; difficult to remix

Example 19 – Inventive Ethylenediaminetetraacetic Acid Suspension

Ethylenediaminetetraacetic acid (EDTA) is a well known chelating agent for metal ions. EDTA is used in a wide variety of applications including agriculture, cleaning products, oilfield,

paper, personal care, and metal working among others. The low water solubility of EDTA (<0.1% at 25°C) makes it difficult to supply as an aqueous solution in the acid form. EDTA is frequently converted to a salt to achieve water solubility, but this is unnecessary if the system pH is acidic and the concentration at use dilution is soluble. 122.5 grams of a EDTA and 7 grams of Rheocin® are dispersed into 221.5 grams pre-thickened polyethylene glycol (200MW) containing 3.5 grams thickening silica, specifically CAB-O-SIL® TS-530 making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
EDTA	35%
Rheocin®	1%
Pre-thickened Polyethylene glycol 200MW	63%
Initial Viscosity	3100 cP
Initial density	1.29 g/ml
Pounds of EDTA per U. S. gallon	3.76

Properties on aging

	Separation	Packing
24 hours	0%	None
7 days	0%	None
1 month	<1%	None
3 months	2%	None

The above compositions are easily pourable or pumpable

Example 20 – Control Ethylenediaminetetraacetic Acid Suspension

This example compares the suspension properties of EDTA to example 19 without the use of the hydrogenated castor wax suspension agent. 129 grams of a EDTA is dispersed into 240 grams polyethylene glycol (200MW) making up the balance in a 600 ml beaker. The mixture is agitated using an overhead mixer at 700 rpm for a period of 1 hour. At this time the viscosity of the mixture is measured on a Brookfield RV viscometer at 20 rpm using a #4 spindle. A portion of the contents is transferred to a 100 ml graduated cylinder for subsequent measurement of the supernatant separation over time. The balance of the material is transferred to another container for evaluation of particle packing and other properties as desired.

Composition:	% by weight
--------------	-------------

EDTA	35%
Polyethylene glycol 200MW	65%

Initial Viscosity	300 cP
Initial density	1.26 g/ml
Pounds of EDTA per U. S. gallon	3.7

Properties on aging

	Separation	Packing
24 hours	43%	Soft packing, difficult to remix
7 days	46%	Hard packing, very difficult to remix
1 month	48%	Hard packing, very difficult to remix
3 months	51%	Hard packing, very difficult to remix

Although the present invention has been described in terms of the foregoing embodiment, such description has been for exemplary purposes only and, as will be apparent to one of ordinary skill in the art, many alternatives, equivalents, and variations of varying degrees will fall within the scope of the present invention. That scope, accordingly, is not to be limited in any respect by the foregoing description; rather, it is defined only by the claims that follow.